EVALUATION OF PHYSICAL-CHEMICAL CHARACTERISTICS OF COMMERCIALLY AVAILABLE FLUORIDE MOUTH RINSES

IULIANA CREȚESCU, RALUCA HORHAT*, OANA MUNTEANU*,#, RODICA CAPRIȚĂ***

*"Victor Babeş" University of Medicine and Pharmacy, 2 Eftimie Murgu Square, 300041, Timişoara, Romania, #e-mail: oanamnt@gmail.com
**"King Michael I of Romania" Banat's University of Agricultural Sciences and Veterinary Medicine from Timişoara, 300654, 119 Calea Aradului Street, Timişoara, Romania

Abstract. A pH value below 5.5 is considered critical for dental erosion due to chemical processes. This paper investigates physical-chemical characteristics of several fluoride mouth rinses available in Romania. We evaluated the following parameters: pH, surface tension, viscosity, titratable acidity, electrical conductivity and total soluble solids (*TSS*) content. Five types of mouth rinses, classified according to their fluoride content, were analyzed in triplicate, and the average values were considered for the statistical analysis. All measurements were performed using standardized equipment and methods, whereas statistical analysis was performed using the XLSTAT software, Version 2015.1. Most mouth rinses turned out to be acidic, but only two types were potentially erosive, with pH < 5.5. The effect of the fluoride content on the physical-chemical characteristics of mouth rinses was found very significant: pH – p-value (two-tailed) = 0.0000, surface tension – p-value (two-tailed) < 0.0001, viscosity – p-value (two-tailed) = 0.001) and for *TSS* content – p-value (two-tailed) = 0.002. Some mouth rinses presented high levels of *TSS* content and viscosity, which may increase their cariogenic and erosive potential. Our study suggests that mouth rinses have the potential to improve oral health provided that the treatment protocol is designed by a dentist taking into account the physical-chemical properties of the mouth rinse of choice.

Key words: mouth rinse, fluoride, pH, viscosity, surface tension, total soluble solids content.

INTRODUCTION

Dental caries represents a frequent condition, affecting around 60–90% of schoolchildren and the vast majority of adults worldwide [15]. They are caused by several factors, such as suboptimal oral hygiene and high intake of fermentable carbohydrates. The effect of these factors is a dynamic imbalance between enamel demineralization and remineralization. Chemical processes are responsible for dental erosion. Loss of tooth enamel occurs at pH ≤ 5.5 [4, 7]. Caries can appear at any person at any age, irrespective of sex or socio-economic status.

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The prevention of caries was and remains a priority of the dental services and it is considered to be more advantageous than their treatment [3, 14].

Fluoride use represents an efficient strategy in preventing and controlling dental caries, and acts like a fluoride reservoir, which is released when the pH of the dental surface drops under 5.5, inducing the remineralization of the tooth enamel [5–9].

Nowadays we use a large variety of cosmetic products, such as mouth rinses that aim to complement the mechanical tools of oral hygiene by targeting the dental biofilm. Mouth rinses with fluoride content are recommended by dentists to their patients with high risk for caries formation [1, 10].

Mouth rinses containing from 0 to 250 ppm fluoride (F^-) ions are available for consumers and are used in many countries. These gained an important market share in the category of dental products as an instrument for improving public health.

Mouth rinses are non-prescription drugs, readily available to children and adults. Nevertheless, the literature shows that there are potential risks/secondary effects of rinsing with such products due to their physical-chemical properties such as pH, titratable acidity, and active substance content. Total soluble solid content is the amount of soluble solid such as sugars, salts, proteins, or acids present in an aqueous solution. Viscosity is also important in the erosion produced by the mouth rinses, since enamel exposure time depends on this parameter [12].

The aim of this study was to evaluate some physical-chemical parameters of fluoride mouth rinses available on the Romanian market.

MATERIALS AND METHODS

MATERIALS

Five types of mouth rinses with sodium fluoride (NaF), classified according to their declared fluoride content, were analyzed in triplicate, and the mean values were considered for statistical analysis. The measured parameters were as follows: pH, surface tension (σ), viscosity (η), specific viscosity (η_{sp}), kinematic viscosity (ν), density (ρ), relative density (*dr*), total soluble solids (*TSS*) content, titratable acidity (*TA*) and electrical conductivity (*EC*). The physical-chemical characteristics were determined using instruments and standardized methods [2]. The analysis of the pH and electrical conductivity (*EC*) were performed at room temperature (26 ºC) using the Consort C3010 electrochemical analyzer (Consort, Belgium) immediately after the bottle was opened. Viscosity was measured using the Ubbelohde viscosimeter at room temperature (26 ºC). Total soluble content readings were performed by refractometry using an Atago refractometer. Surface tension was measured by the stalagmometric method. Titratable acidity was determined by titration of a known amount of mouth rinse with 0.1 N NaOH, using phenolphthalein as an indicator [2].

STATISTICAL ANALYSIS

All measurements were performed at least in triplicate. The values of the measured physical-chemical parameters were expressed as the mean ± standard deviation (SD), at a confidence level of 95%. Data were analyzed using the statistical analysis software XLSTAT, Version 2015.1. Student's t-test was used to demonstrate statistically significant differences between various mouth rinses. To quantify relationships between the investigated variables, the values of the Pearson correlation coefficient, r, were interpreted as follows [11]: very strong correlation for $r \geq 0.7$; substantial correlation for $0.5 \leq r < 0.7$; moderate correlation for $0.3 \leq r < 0.5$, and low correlation for $0.1 \leq r < 0.3$. A p-value < 0.05 was considered statistically significant.

RESULTS

Table 1 lists the measured physical-chemical parameters of mouth rinses included in this study, classified according to their fluoride content.

Table 1

The physical-chemical parameters (mean±SD) of mouth rinses of different fluoride contents

Parameter / Fluoride content of the sample (ppm)	0	90	217	225	250
pH	5.47±0.006	5.28±0.02	6.81±0.01	6.24±0.93	6.53±0.53
EC[mS/cm]	18.42±1.49	2.315±0.005	26.55±1.75	7.092±7.39	10.8295±7.29
dr	1.0714±0.0002	1.0201±0.0005	1.7850±0.41	1.0527±0.016	1.1594±0.23
ρ[g/cm^3]	1.0698±0.0002	1.0187±0.0006	1.077±0.0003	1.0518±0.017	1.0455±0.004
σ[dyn/cm]	45.84±0.0000	34.64±0.34	39.31±0.48	34.66±3.11	36.06±2.018
η[mPas]	1.4569±0.003	1.9452±0.06	1.8559±0.04	2.0123±0.93	1.4953±0.32
η_{sp}	0.6675±0.003	1.2264±0.06	1.1242±0.045	1.3032±1.07	0.7115±0.36
v[cm^2/s]	1.3618±0.003	1.9096±0.05	1.7232±0.036	1.9081±0.86	1.4294±0.30
TSS[%]	13.04±0.29	11.16±0.24	14.57±0.27	12.19±2.88	9.525±2.96
TA	2.927±0.26	1.8215±0.025	2.566±0.017	1.2975±1.11	1.2648±0.67

The effect of fluoride content on the physical-chemical characteristics of mouth rinses is very significant (pH – p-value (two-tailed) = 0.000, surface tension – p-value (two-tailed) < 0.0001, and viscosity – p-value (two-tailed) = 0.001) and for *TSS* content – *p*-value (two-tailed) = 0.002 and *TA* *p*-value (two-tailed) = 0.011).

The rinse that showed the lowest pH value (5.28) had a higher value of the kinematic viscosity (1.9096 cm^2/s) and the rinse with the highest pH (6.81) had a relatively high kinematic viscosity (1.7232 cm^2/s). The rinse with the lowest kinematic viscosity (1.3618 cm^2/ s) also had a relatively low pH, under the critical limit (5.47).

Table 2 presents the Pearson correlation coefficients between the investigated physical-chemical characteristics of the mouth rinses included in this study. Here, extremely strong positive correlations ($r \geq 0.9$) are highlighted by bold type. The correlation coefficients listed in Table 2 indicate a wide range of correlations, from a very strong positive correlation to a very strong negative one [11] (for their interpretation, see the last paragraph of Materials and Methods).

Table 2

Pearson correlation matrix between physical-chemical characteristics of mouth rinses‡

	pH	*EC* [mS/cm]	*dr*	ρ [g/cm³]	σ [dyn/cm]	η [mPa s]	η_{sp}	*v* [cm²/s]	*TSS* [%]	*TA*
pH	**1**	0.518	0.712	0.511	–0.202	0.040	0.040	–0.051	0.165	–0.228
EC [mS/cm]	0.518	**1**	0.821	**0.921**	0.663	–0.326	–0.326	–0.442	0.736	0.710
dr	0.712	0.821	**1**	0.632	0.152	0.108	0.108	0.008	0.648	0.394
ρ [g/cm³]	0.511	**0.921**	0.632	**1**	0.688	–0.315	–0.315	–0.447	0.726	0.607
σ [dyn/cm]	–0.202	0.663	0.152	0.688	**1**	–0.653	–0.653	–0.709	0.513	0.876
η [mPa s]	0.040	–0.326	0.108	–0.315	–0.653	**1**	**1.000**	**0.990**	0.244	–0.313
η_{sp}	0.040	–0.326	0.108	–0.315	–0.653	**1.000**	**1**	**0.990**	0.244	–0.313
v [cm²/s]	–0.051	–0.442	0.008	–0.447	–0.709	**0.990**	**0.990**	**1**	0.130	–0.372
TSS [%]	0.165	0.736	0.648	0.726	0.513	0.244	0.244	0.130	**1**	0.748
TA	–0.228	0.710	0.394	0.607	0.876	–0.313	–0.313	–0.372	0.748	**1**

‡Values in bold type highlight correlation coefficients larger than 0.9.

There is a high positive correlation (*r* = 0.518) between the pH and the electrical conductivity and also between the *TA* and the electrical conductivity (*r* = 0.710). It should be mentioned that the electrical conductivity of a mouth rinse is not influenced only by the fluoride content, but results from its total chemical composition. Between the pH and *TA* there is a moderate negative correlation (*r* = –0.228).

According to the Pearson correlation matrix (Table 2), the measured physical-chemical quantities are correlated. Therefore, we performed principal components analysis (PCA) of the set of measured variables. PCA, the simplest of the true eigenvector based multivariate analyses, is a factor analysis technique. Its goal is to reduce the number of variables a phenomenon depends on, identifying a set of representative variables that still contains most of the information enclosed in the large set of variables. Using PCA, we identified two independent (uncorrelated) factors, F1 and F2, expressed as linear combinations of the original variables, which retained most of the information contained in the original variables (Fig. 1).

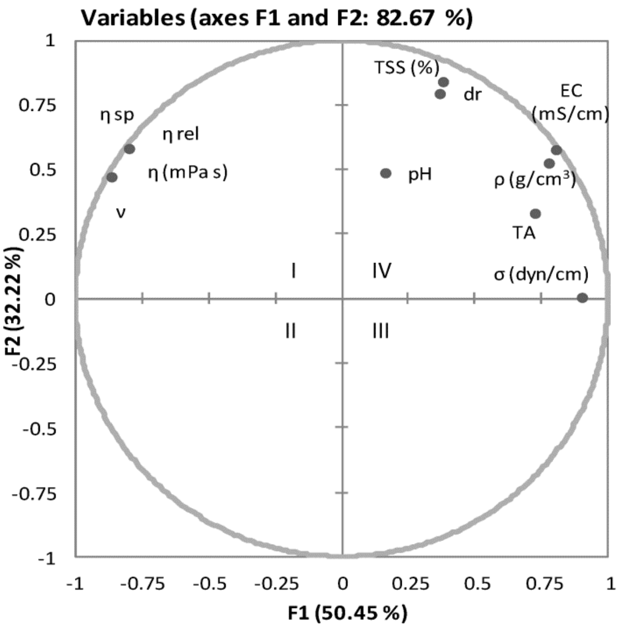

Fig. 1. Principal components analysis of the investigated physical-chemical characteristics.

The density, electrical conductivity, pH, *TA* and *TSS* content of the samples are grouped in the fourth quadrant, the kinematic viscosity and the dynamic viscosity are grouped in the first quadrant, which shows the presence of a significant correlation, as also shown in Table 2.

DISCUSSION

The integrity of the tooth enamel hinges on a dynamical equilibrium between demineralization and remineralization. The mineral component of the tooth enamel is hydroxyapatite, $Ca_5(PO_4)_3OH$. During demineralization, hydroxyapatite dissociates, releasing calcium, phosphate and hydroxide ions. During remineralization, these ions reconstitute the hydroxyapatite crystal provided that their ionic product, $[Ca^{2+}]^5[PO_4^{3-}]^3[OH^-]$, is larger than the solubility product of hydroxyapatite, as usual in fresh saliva at pH 7. As the pH drops, however, so does the hydroxide ion concentration and the phosphate ion concentration (due to protonation). Below a critical pH, of about 5.5, demineralization becomes dominant [10]. Fluoride ions are beneficial to the enamel in two ways [8, 9]: (i) by raising the pH and (ii) by facilitating remineralization in the form of fluorapatite, $Ca_5(PO_4)_3F$.

The effect of fluoride content on the physical-chemical characteristics of mouth rinses was found very significant. Some mouth rinses presented high levels of *TSS* content and viscosity, which, used improperly in terms of frequency and timing, might increase their cariogenic and erosive potential.

Some aromatic substances and sweeteners are added in order to enhance the taste of mouth rinses, especially for children. The cariogenic potential is directly linked to the high level of sugar content in formulas. There are studies which found the lowest content of *TSS* (7.0%) and the highest value of *TSS* (22.5%). The content of *TSS* in fluoride free mouth rinses for children was 13.04% and the highest level of 217 ppm (14.57%) was found in those with fluoride [4]. In the literature there is no information related to the maximum admitted value of TSS in mouth rinses.

The viscosity is controlled by the internal friction forces between adjacent liquid layers. Viscosity was measured at room temperature of 26 °C. Taking into account that the temperature of the human body is around 37 °C, our results are slightly higher than the viscosities of these mouth rinses in the oral environment. Nevertheless, the hierarchy of viscosities observed here (Table 1) is expected to remain the same also at physiological temperatures.

The work by Dwitha *et al*. [6] emphasizes the importance of various physical-chemical properties of saliva, such as salivary flow rate, pH, buffering capacity and viscosity, which serve as markers for estimating the risk of dental caries formation.

The pH in mouth rinsing solutions ranged from 5.28 to 6.81. It is known that the erosive process could not be assigned only to pH values. In this regard, it was decided to measure the viscosity. There are few topics in the literature related on viscosity values. In our study, for the lowest value of the pH (5.28) we get the highest value of the kinematic viscosity (1.9096 cm^2/s).

The titratable acidity is directly related to the buffering capacity of the saliva; samples with low titratable acidity are readily neutralized by oral fluids [8].

Although most mouth rinses displayed a low level of acidity, two types were potentially erosive (with pH less than 5.5). Nevertheless, there are reasons for the acidity of mouth rinses. The low pH increases the chemical stability and favors the incorporation of fluoride ions into the enamel, making it less soluble and more resistant to acid attack. In the presence of fluoride, phosphate and calcium ions, low pH favors the precipitation of calcium fluoride on the tooth surface, reinforcing the dental enamel with a fluorapatite layer [8]. Another justification of mouth rinse acidity is that low pH decreases the biofilm metabolism compared to the glycolytic method (fermentation), and produces extracellular polysaccharides. Despite these benefits, the literature shows that a very low pH (3.7) associated with the absence of fluoride ions is harmful for the dental enamel, leading to demineralization [4, 10, 13].

CONCLUSIONS

This paper reports physical-chemical characteristics of mouth rinses (density, viscosity, surface tension, pH, electrical conductivity, total soluble solids content, and titratable acidity), and discusses their potential impact on oral health.

Our study shows that there is a high positive correlation (r = 0.518) between pH and the electrical conductivity and also between *TA* and the electrical conductivity (r = 0.710). The density, electrical conductivity, pH, *TA* and *TSS* content of the samples are grouped in the fourth quadrant, which shows the presence of a significant correlation, as is also shown in Table 2 and Fig 1.

In light of the above, mouth rinses should be used according to the advice and under periodic surveillance of a dental practitioner. In order to optimize the oral health outcome, treatment parameters, such as the rinse frequency and duration, should be established by a dentist. This is especially important for children, who might be influenced by the taste of the mouth rinse, or by incentives provided by their parents.

Our work suggests that the demineralization potential of mouth rinses deserves further investigation *in vitro* and *in vivo* because some physical-chemical factors, such as salivary flow and buffering capacity, might influence their overall effect on the tooth enamel.

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